



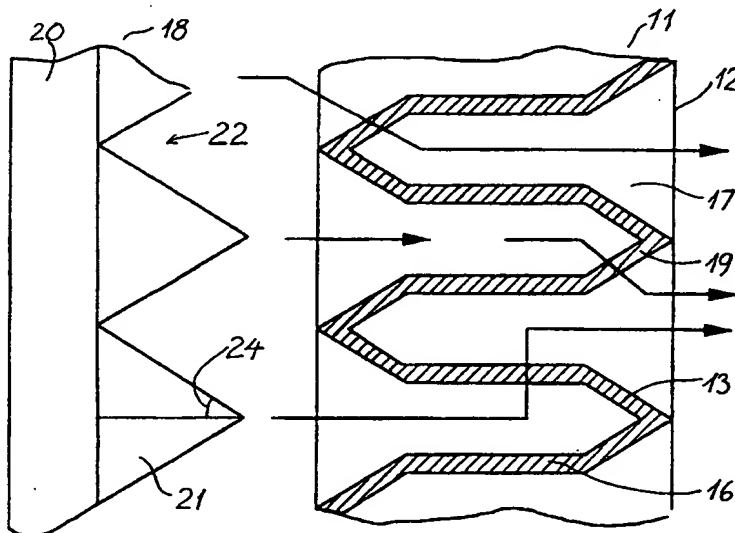
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(54) Title: A METHOD FOR CLOSING A PASSAGE IN A FILTER BODY SAMPLE



(57) Abstract

A method for closing a passage in a filter body sample made from a solidifiable plastic or plastifiable particle-based material and comprising a plurality of coextending throughgoing passages separated by common passage walls, the method comprising deforming passage walls adjacent to the passage. This method vastly simplifies the manufacturing of filter bodies for use in e.g. filtering flue gasses or substrates for holding a catalyst. The filter body is preferably made of SiC. Producing a filter body for filtering flue gas may comprise: producing from a solidifiable plastic or plastifiable particle-based material, a filter body sample comprising a plurality of coextending throughgoing passages separated by common passage walls; closing one end of substantially each passage by deforming passage walls adjacent to the passage, and; subsequently solidifying the plastic or plastifiable particle-based material to a porous solid material.

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A Method for Closing a Passage in a Filter Body Sample

This invention relates to improvements in connection with particle-based filter bodies for removing fine solid particles from fluids, in particular for removing fine solid particles from gases, such as for removing soot particles from exhaust gas from diesel engines.

In operation, such filters will be exposed to quite hostile environments; thus, the filters should be able to withstand high temperatures and the corrosiveness of the exhaust gases. Soot particles generated in the diesel engine combustion process are of a size down to about $0.1\text{ }\mu\text{m}$; however, they have quite sticky surfaces and tend to agglomerate to larger particles of the order of $1\text{-}5\text{ }\mu\text{m}$ in the exhaust pipe outside the combustion chamber. This means that the filters should be able to remove particles in the size from about $0.1\text{ }\mu\text{m}$ to about $10\text{ }\mu\text{m}$. The use of the filter bodies on vehicles will pose certain constraints on the design of the filter bodies, as they should be able to withstand shocks and vibrations due to movements of the vehicle, and as the dimensions of the filter should be as small as possible.

A type of filter body which has been found useful for the above purpose is made of a porous ceramic, metallic and/or metal-like material which is able to withstand the hot corrosive exhaust gases. The shape of the filter body is typically designed so that the gas has to travel through walls of the porous material, the walls having a porosity and a pore size so that the soot particles are not able to travel through the wall, whereby the gas is filtered. The porosity and the thickness of the filtering walls are chosen so as to provide a suitable filtering efficiency and an acceptable pressure drop over the filter body.

Filter bodies of this known type may be manufactured by extruding a plastic paste of a particle-based material such as a ceramic, a material based on particles of a metal-like

materials, such as SiC, or a material based on particles of a metal, such as stainless steel, into the desired cross-sectional shape of the filter body. This shape preferably comprises a honeycomb-like configuration, so that the extruded body contains a number of parallel passages extending along and all through the length of the body from an inlet side of the filter body to an outlet side, the passages being separated by common passage walls. After the extrusion, the known method of production comprises blocking all passages by means of plugs of the same plastic material as the filter body ("plugging") in at least one end of each passage to prevent that during operation, the gas flows directly through the filter body without having been filtered by travelling through a porous wall of the filter body. Thus, in a filter body of this kind, the gas travels into the passages open at the inlet side, through the walls into the passages open at the outlet side and then out of the filter body.

After plugging of the passages, the filter bodies are sintered to form the final solid filter body. Filter bodies made in this way are disclosed in European Patent Specification No. 0 089 756 and in published European Patent Application No. 0 336 883.

The filtering efficiency of a filter body of this known type is defined by the dimensions of the filter body and the characteristics of the sintered material, such as the pore size and the porosity thereof. Those dimensions of the filter body which contribute to defining the filtering efficiency of the filter body are the total area and the thickness of the filtering walls in the filter body. The thickness of the filtering walls of the filter body is defined by the dimensions of the extrusion die. The total area of the filtering walls likewise depends on the dimensions of the extrusion die and on the length of the filter body, but also on the manner in which the channels of the filter body have been closed. When plugs are inserted into the passages, part of the filtering wall will become blocked and, thus, no longer takes

part in the filtration of the flue gas. Thus, plugging a typical filter body having the dimensions \varnothing 170 mm L 250 mm, the passages being quadratic and having side lengths of 2.5 mm and a wall thickness of 1 mm, with 1 cm long plugs will
5 result in blocking of approximately 5% of the total filtering surface of the filter body and, thus, in an increase in the pressure drop over the filter body as part of the porous material therein is now blocked. This increase in pressure drop may be compensated for in two ways. One way is to
10 increase the area of the filtering walls by increasing the width or the length of the filter body and, thus, resulting in a larger filter body, which is not technically or commercially desirable. The other way is to increase the porosity and/or pore size of the filtering material; this is not
15 desired as it will decrease the filtering efficiency of the filter body.

Another problem associated with this known plugged type of filter body is that as the sintered filter body is exposed both to gas pressure and thermal cycling, the plugs may
20 loosen from the rest of the filter body and subsequently fall out from the filter body, resulting in a drastic reduction of the filtering efficiency. Even though it is attempted to avoid this loosening by using quite long plugs, such as of the order of 1 cm as mentioned above, the loosening problem
25 may not be completely solved thereby. The problem with the thermal cycling is especially severe in filter bodies having different thermal expansion coefficients in the direction of the extrusion and in the transverse direction, such as Cordierite (see European Patent Specification Publication No. 0
30 089 756). This may result in stress in the interfaces between the plugs and the extruded material, which again may result in loosening and removal of the plugs. The problem also potentially exists in filter bodies made of SiC (see European Patent Application Publication No. 0 336 883), however, as
35 SiC has the same thermal expansion coefficients in all directions, the resulting stress is not as severe when using SiC as when Cordierite is used.

The actual plugging of an extruded body is typically performed semi-manually, and this step of the manufacturing process is, therefore, typically the most expensive (See e.g. US-A-4,662,911: "Especially, the step of closing the through
5 holes alternately is very difficult"). Thus, it would be highly desirable to obtain a plugging method which may be easily automated and which avoids the problem associated with potential loosening of the plugs.

An alternative way of manufacturing the filter bodies is by
10 shaping these in moulds having a shape being the inverse of that of the desired filter body. Methods of this type may be seen in US-A-4,662,911 and EP-A-0 206 250. However, as only filter bodies with one shape may be made from a mould of this type, this manner of shaping the filter bodies is not pre-
15 ferred compared to e.g. the extrusion process and the versatility thereof.

Thus, an object of the invention is to provide an improved method for closing passages in filter bodies of the above-defined type and to provide, in an economic way, filter
20 bodies having improved properties.

In a first aspect, the invention relates to a method for closing a passage in a filter body sample made from a solidifiable plastic or plastifiable particle-based material and comprising a plurality of coextending throughgoing passages
25 separated by common passage walls, the method comprising deforming passage walls adjacent to the passage.

Thus, the main principle of this aspect of the invention is to avoid relying on the insertion of additional material into the filter body sample, but rather to close the passage by
30 means of material already present in the form of parts of passage walls adjacent to the passage. This, of course, does not mean that it is not possible to add material in connection with the method of the invention, but this is not presently preferred.

The term filter body sample, as used in the present specification and claims, indicates a state of the filter body which is not the final hard, solid state in which the filter body is used for its purpose, but rather a state in which at least the wall parts in question are plastically deformable. As appears from the following detailed description, the wall parts which are to be deformed may be wall parts of a relatively stiff filter body sample, the relevant wall parts being softened prior to the deformation. Typically, the later solidification of the filter body sample in which the passage (or normally all the passages) has/have been closed by deformation of wall parts will take place by sintering of the particle-based material.

In the present context, the term "solidifiable plastic or plastifiable particle-based material" designates a material the main component of which consists of particles. One example of such a material is a ceramic material, typically a plastic clay or clay-like material. Other examples are materials based on metal or metal-like particles and typically a binder system comprising a "green" binder which will bind the system in its plastic or plastifiable state and a ceramic or inorganic binder, e.g. very fine metal or metal-like material, which will bind the material in its final solidified, e.g. sintered, state. Examples of such materials are given in the following. The filter body sample material will be in a plastic or plastifiable state during the deformation of the wall parts, at least the wall parts being deformed being in a plastic, in other words, plastically deformable, state during the deformation.

The deformation of the walls is suitably performed by introducing the walls into a female forming tool forcing the outermost parts of the walls together. Thus, the deformation is performed at an end of the passage, by pressing the female tool on to the end of the filter body sample. As will be easily understood from the drawings and the appertaining text, the female tool is advantageously defined by parts of

adjacently positioned male forming tools positioned on the base of a mould. These male forming tools may suitably be positioned so that each male forming tool is pressed into an individual passage coextending with the passage being closed.

- 5 It is preferred that the passage being closed and the individual passages into which the male forming tools are introduced have in common the walls which are deformed. Thus, when a female forming tool is defined between the male forming tools introduced in the individual passages, the common walls
10 will be introduced in the female forming tool and thus deformed to close the passage to be closed.

Normally, the tool is designed so that several of the passages in the filter body sample can be closed in the same operation.

- 15 It is preferred that the cross-sectional arrangement of the passages is in a number of substantially parallel rows, and that the passages in each row are arranged substantially equidistantly. It is especially preferred that the cross-sectional arrangement of the passages is in a regular pattern,
20 and that the individual passages have substantially identical polygonal cross-sections. Firstly, this is the preferred arrangement already in the known filter bodies, and secondly, such regular arrangement will, of course, facilitate closing of several passages in one operation using
25 suitably designed tools with several systematically positioned mould parts, such as illustrated in the following. In one embodiment, the male forming tools may be positioned on a substantially plane base whereby the deformation of the passage walls is performed substantially simultaneously by
30 all female forming tools defined on the mould. In this way, all male deforming tools are pressed into the end of the filter body at the same time and, thus, the walls to be deformed are all introduced into the female deforming tools at the same time.

- In a second embodiment, the base on which the male forming tools are positioned is shaped substantially as part of a curved surface, preferably a circular drum or wheel which, when rolled over the filter body sample, sequentially introduces the male forming tools in the passages, whereby the walls are deformed sequentially. Thus, as the drum or wheel is rolled over the end of the filter body, the male deforming tools are sequentially introduced into the passages, whereby the deformation takes place sequentially.
- 10 The plurality of passages which are closed may be divided into first and second groups, the passages of the first group being closed at a first end of the filter body sample and the passages of the second group being closed at an opposite second end thereof. This means that the resulting filter body
- 15 will have the above-mentioned preferred flow path configuration where the gas is able to flow into one of the groups of passages open at a gas inlet surface, the gas then to flow through the filtering walls inside the filter and into the passages of the other group open at an opposite gas outlet
- 20 surface and out of the filter body. In this manner, the area of the filtering wall of the filter body is close to being half of the total area of all passage walls which are common to passages of different groups. If all channels were closed at the same end of the filter body, the total filtering
- 25 surface would be the cross-sectional area of the gas inlet side surface of the filter body; the filtering walls inside the filter body would not take part in the filtering of the flue gas. This is the reason why the well-known checkerboard pattern is preferred also for the filters made according to
- 30 the present invention and, indeed, can be utilized to its maximum potential because the dead area caused by the plugs in the known filters is substantially avoided.

In a second aspect, the invention relates to a method for producing a filter body for filtering flue gas, the method

35 comprising

- producing, from a solidifiable plastic or plastifiable particle-based material, a filter body sample comprising a plurality of coextending throughgoing passages separated by common passage walls,
- 5 - closing one end of substantially each passage by deforming passage walls adjacent to the passage, and
- subsequently solidifying the particle-based material.

For simplicity of the manufacturing of the filter body sample and for the common walls to be as efficient in the filtering as possible, it is preferred that the cross-sectional shape of the through-going passages are polygons and that the male forming tools have a cross-section being a polygon of the same order as the polygons of the cross-sectional shape of the through-going passages. When the passages have the cross-sectional shape of polygons, the sides thereof may be constituted by the common filtering walls, whereby these common filtering walls may have equal thickness in substantially all of the filter body; this enables substantially all parts of the filtering wall in the filter body to take equal part in the filtering of the flue gas. This minimizes the pressure drop over the filter body as well as it offers a filter body being able to filter large amounts of flue gas before being fully clogged by the soot deposited in the filter body.

The cross-sectional polygon may be of order 4, whereby it is preferred that at least the tip of the male forming tools are of the shape of a pyramid.

A most interesting type of particle-based material is a material based on particles of SiC, which material can be shaped by extrusion, subjected to the method according to the invention for closing passages, and then sintered.

The mean pore size of the final porous SiC filter body will normally be in the range of 1 to 150 μm , in particular 10-100 μm . The preferred sub-ranges within this range will depend on the particular separation to be performed, in

particular on the size of the particles to be retained by the filter. Thus, for removal of soot particles from diesel exhaust gas, the pore size of the material will normally be in the range of 15-80 μm , such as 20-50 μm . The higher the mean pore size, the lower the pressure drop over the filter body will be. One of the advantages of filter bodies made from particle-based SiC materials is that they can be made with a relatively high thermal conductivity, such as at least 5 W/mK and typically in the range of 5-30 W/mK, such as 5-15 W/mK. A high thermal conductivity will result in a high thermal shock resistance and thus resistance towards thermal shocks which may occur when the soot collected in the filter body is burned off, which is the normal way to purify and regenerate the filter body.

The porosity of the particle-based material, whether a SiC material or not, significantly influences the strength of the material. Thus the porosity selected will depend on the required physical strength properties, the selected pore size of the filter body and the particles to be filtered, and will normally be in the range of 30-90%, typically in the range of 40-75%.

Particulate SiC will normally have a weight average mean particle size in the range of 10-250 μm , such as in the range of 20-150 μm , preferably in the range of 30-100 μm . The selected particle size and particle size distribution will normally be dictated by realistic available commercial grades and the pore sizes and other properties aimed at in the final material. Seven commercial grades of SiC are presently preferred starting products for producing the porous material. One grade is particulate SiC of mesh 120, substantially corresponding to a particle size range of 88-125 μm . Another grade is particulate SiC of mesh 150, substantially corresponding to a particle size range of 63-105 μm . A third preferred grade is particulate SiC of mesh 180, substantially corresponding to a particle size range of 53-88 μm . A fourth preferred grade is particulate SiC of mesh 220, substantially

corresponding to a particle size range of 44-74 μm . A fifth preferred grade is particulate SiC of mesh 280, substantially corresponding to a particle size range of 35-38 μm . A sixth preferred grade is particulate SiC of mesh 320, substantially
5 corresponding to a particle size range of 28-31 μm . A seventh preferred grade is particulate SiC of mesh 360, substantially corresponding to a particle size range of 21-28 μm .

In addition to a large amount of particulate SiC having a particle size in the ranges described above, the material to
10 form the basis of the preferred SiC-based materials typically comprises a smaller amount (up to 15 %wt of the total weight) of a fine-grained (typically in the order of 0.3-2 μm) sintering additive such as SiC and/or SiO_2 and/or carbon black, a binder, such as cellulose ether, water, an alcohol
15 such as ethanol and optionally poly vinyl alcohol, a lubricant and/or a plastifier.

The shaped body or sample of paste may then be dried in a controlled atmosphere to confer some handling strength there-
to while still preserving the possibility of keeping or
20 making the walls deformable. Often, it will be preferred to dry the sample to a relatively high handling strength and then to soften the wall ends by soaking with a suitable liquid such as water or a mixture, such as an emulsion, of water and oil, thereby obtaining a plasticity of the wall
25 ends which is suitable for the deformation performed according to the invention. After the deformation to close the passages, the sample is again dried, and then, the dried sample is placed in a high temperature oven where the temperature is elevated to temperatures in the order of 300-
30 500°C whereby the binder is burned away, giving the SiC sample a rigid, open structure. Due to the smaller radii of curvature the fine-grained sintering additive, these small particles will evaporate when further elevating the temperature to in the order of above 2200°C up to about 2600°C. When
35 evaporating, this material will condense at the grain contacts of the larger SiC particles and hence enhance the

physical strength and the thermal conductivity of the sintered material; also the electrical conductivity of the material will increase hereby.

Even though the presently preferred material is SiC, the present invention may be used in the manufacture of filter bodies made of a wide variety of other particle-based materials such as B_4C , $BaTiO_3$, Si_3N_4 , BN, Al_2O_3 , Mullite ($3Al_2O_3 \cdot 2SiO_2$), Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$), Cordierite combined with β -spodumen ($Li_2O \cdot Al_2O_3 \cdot 4SiO_2$), β -SiC, Stabilised ZrO_2 , $ZrO_2 + TiO_2$, TiC-TiN- Al_2O_3 combinations, TiC- Al_2O_3 combinations, Na_xWO_3 ($0 < x < 1$), Al_2O_3 silicates, MgO silicates, CaO, silicates, Titanium oxide or M_2B , MB, M_2B_5 , M_2C , MC, M_2N , MN, M_3Si , M_3Si_2 , M_5Si_3 or MSi_2 , where M is Mo or W.

In the following, the invention will be described in greater detail with reference to the figures, wherein

Fig. 1 illustrates a cross-sectional view of an extruded filter body plugged according to the prior art,
Fig. 2 is a partial cut-away side-view of the prior art filter body seen in Fig. 1,
Fig. 3 is a cross-sectional view of a filter body manufactured by a first embodiment of the method according to the invention and of a mould for use in the embodiment,
Fig. 4 is a perspective partly cut-away view of an end of a filter body made according to the first embodiment of the method of the invention,
Fig. 5 illustrates a mould for use in the first embodiment of the method of the invention,
Fig. 6 is a cross-sectional view of a filter body manufactured by a second embodiment of the method according to the invention and of a mould for use in the embodiment,
Fig. 7 schematically illustrates how the passage walls are deformed in this second embodiment of the method of according to the invention,

Fig. 8 illustrates an alternative embodiment of a mould for use in the method of the invention,

Fig. 9 illustrates another alternative embodiment of a mould for use in the method of the invention,

5 Fig. 10 illustrates the second embodiment of Fig. 6 wherein a shape-preserving body has been inserted, and

Fig. 11 illustrates a comparison of flow characteristics between a filter body plugged according to the prior art and a filter body closed according to the method of the inven-

10 tion.

Fig. 1 is a schematic cross-sectional view of a filter body 1 comprising an extruded body 2 having a number of through-going passages 7 extending from a gas inlet side surface 4 to a gas outlet side surface 5, with plugs 3 according to the
15 prior art. Each end of the filter body 1 is plugged in a "checkerboard" pattern so that all blind passages open at the inlet side surface 4 of the filter body 1 have filtering walls 6 common only with passages open at the outlet side 5 surface of the filter body 1. This structure is the so-called
20 honeycomb filter structure.

As appears from this figure, the outermost parts 9 of the filtering walls 6 are blocked by the plugs 3. In practice, about 5% of the total area of the filtering walls 6 will typically be blocked in this type of filter body 1. The
25 arrows show the flow of flue gas from the gas inlet side surface 4 through the filter body 1 to the gas outlet side surface 5. It is seen that as the length of the plugs is larger than the thickness of the filtering walls 6 (typically in the order of a factor of 5), and as the material of the
30 walls 6 and the plugs 3 is often the same, the gas will not flow through the plugs 3, whereby the total filtering surface of the filter body 1 is reduced by the plugs 3.

Fig. 2 illustrates the filter body 1 discussed in connection with Fig. 1. It is seen that the flue gas inlet side surface
35 4 and the gas outlet side surface 5 are opposite and that a

first plurality 8 of blind filtration passages extends transversely into the filter body 1 from the inlet side surface 4 and that a second plurality 10 of blind filtration passages extends transversely into the filter body 1 from the outlet side surface 5. It is further seen that the blind filtration passages of the first 8 and second 10 pluralities are arranged spaced apart and mutually juxtaposed in the filter body 1.

Figs. 3 and 4 illustrate a filter body 11 manufactured according to a first embodiment of the method according to the invention. It is seen that some of the original through-going passages 17 of the extruded body or filter body sample 12 are closed by deforming each end of the extruded body 12 by pressing a mould 18 into the end of the extruded body 12. During this closing, outermost parts 19 of the filtering walls 16 in that end are deformed by female forming tools 22 of the mould 18.

The female forming tools 22 of the mould 18 are defined by male forming tools or pyramid-shaped elements 21, positioned on a base 20 of the mould 18, the cross-sectional shape of which is chosen so as to ensure sufficient deformation of the outermost parts 19 of the filtering walls 16 so as to close at least some of the through-going passages 17 of the extruded body 12 when the mould 18 is pressed into an end thereof.

In this embodiment, a honeycomb-shaped filter body is manufactured, which means that the passages 17 have quadratic cross-sections and that all blind passages being closed at one end have common walls only with blind passages being closed at the other end. As the deformation performed on both ends of this filter body is identical, except for a displacement of the mould 18 so as not to close the passages already closed in the other end and thus to have passages that are not closed at all, the same mould 18 may be used in both ends of the honeycomb-shaped filter body 11.

Fig. 3 illustrates a principle of the invention that the outermost parts 19 are deformed to close a passage by introducing walls adjacent to this passage to be closed into a female forming tool 22 which performs the closure. It is seen that the walls are introduced in the female forming tool 22 by introduction of the pyramid-shaped elements 21, which define the female forming tool 22, in at least some of the passages adjacent to the passage to be closed. By choosing pyramid-shaped elements 21 with suitable cross-sections and suitable positions on the base 20, a suitable closure of the passage may be obtained.

It is clear that a passage into which a pyramid-shaped element 21 is introduced in one end, can not be closed at that end, that is, at least not in the same operation. In this embodiment, the pyramid-shaped elements 21 are positioned on the base 20 so as to be introduced in all passages covered by the base 20 and which are not to be closed at the end to be deformed. Thus, all four passages adjacent to a passage to be closed will receive pyramid-shaped elements 21 in order to produce a honeycomb-shaped filter body 11.

The mould 18 shown in Fig. 5 may be used for this purpose. The deformed end of a filter body 11 deformed by the mould 18 of Fig. 5 is shown in Fig. 4. It is seen that when the pyramid-shaped elements 21 are to be introduced in the passages not to be closed, the elements 21, even though both the passages and the elements 21 have quadratic cross-sections, are rotated an angle of in the order of 45° so as to enable the elements 21 of the mould 18 to be introduced in the correct passages. This is seen from Fig. 4. In this manner, the female forming tools 22 deforming the outermost parts 19 of the walls 16 are defined by four elements 21; this may be realized from studying Fig. 4 where it is seen that the closed passages are positioned under the "crosses" 23 of the deformed outermost parts 13 of the filtering walls 16.

It may be preferred to perform the deformation in several steps with a number of moulds 18 having pyramid-shaped elements 21 positioned in the same positions on the base 20 but having different angles 24, starting with small angles, and thus small deformations not being sufficient to perform closure of the passages, and finishing with large angles, so as to obtain a closure of the passages.

As mentioned above, it may be preferred to soften the end of the extruded body 12 before deformation, especially if the extruded body 12 has been dried to increase the handling strength thereof. This softening may e.g. be obtained by soaking the end of the extruded body in a soaking liquid such as explained in greater detail below.

To ensure that the closures are gas-tight, the material of the deformed wall parts 13 is preferably mildly "kneaded" or pressed together by rolling a roller, preferably a steel cylinder, over the ends of the filter body 31 after deformation.

When using this method for producing the blind passages, the total area of the filtering walls 16 in the filter body 11 is not significantly reduced as also the deformed parts 13 of the filtering walls 16 take part in the filtering of the gas. This is illustrated by the arrows in Fig. 3, showing the flow of gas from a gas inlet side surface 14 through the filter body 11 to a gas outlet side surface 15, also going through the deformed parts 13 of the filtering walls 16.

It should be noted that the deformation of the filtering walls 16 does not significantly alter the filtering characteristics of the material of the deformed parts 13 compared to the characteristics of the walls 16.

Furthermore, it is seen that the deformed parts 13 of the filtering walls 16 give the filter body 11 funnel-shaped gas entrances and gas exits which give a better flow of gas

through the filter body 11 and, thus, further reduce the pressure drop over the filter body 11.

If the filter body 11 is not to be honeycomb-shaped, such as if it was desired to have a majority of passages closed in one end of the filter body 11, it might not be possible to use the same mould 18 in both ends of the extruded body 12. In this case two different moulds - one for use in deforming each end of the extruded body 12 - may be used.

Fig. 6 illustrates a second embodiment of the method according to the invention. In this figure, as in Figs. 3 and 4, a filter body 31 comprises an extruded body 32 having a number of through-going passages 37. Also in this embodiment a honeycomb-shaped filter body 31 is manufactured. Therefore, the extruded body 32 comprises a plurality of through-going passages 37 being separated by filtering walls 36. These passages 37 are preferably positioned in a pattern wherein the passages are positioned in rows. Due to the honeycomb shape of the filter body 31, every second of the passages in a row are closed at one end and the other half are closed at the other end.

In this embodiment, the deformation is performed by introduction of the outermost parts 39 of the walls 36 into female forming tools 40 defined by teeth 41 positioned on a wheel 38 in a manner so that when one tooth is pressed into a passage of the filter body 31 and when the wheel is rotated and translated, the next tooth will fit into another passage of the filter body 31. Preferably, the wheel 38 is orientated and rotated so that the teeth 41 of the wheel 38 enter every second passage 37 in a row and thereby closes the passages positioned between two passages in which teeth 41 are introduced.

To ensure a sufficient closure of the passages, the depth of the teeth 41 (into the plane of the figure) is preferably

substantially identical to the width of the passages 37 (also into the plane of the figure) of the filter body 31.

The function of the wheel 38 of this second embodiment will now be described in detail with reference to Fig. 7, wherein
5 the wheel 38 has been displaced upwardly for better illustrating the function thereof.

The scenario is as follows: three passages are considered which, in the direction of the wheel 38 (see the arrow), are: a first 51, a second 52 and a third passage 53. A first wall
10 71 is positioned between the first 51 and the second passage 52 and a second wall 72 is positioned between the second 52 and the third passage 53.

The first passage 51 is the first passage to receive a first tooth 61 and the third passage 53 is to receive a second
15 tooth 62 of the wheel 38, the second tooth 62 of the wheel 38 being the tooth adjacent to the first tooth 61 in the direction of movement and rotation of the wheel 38 (see the arrow).

The second passage 52 is to be closed by introducing outermost parts 81 and 82 of the walls 71 and 72, respectively,
20 into a female forming tool 55 defined by the first 61 and second teeth 62 of the wheel 38 whereby the deformed outermost parts 81 and 82 of the first and the second walls 71 and 72, respectively, are to form the closure of the second
25 passage 52 after the deformation.

Due to the first tooth 61 being introduced in the first passage 51 and due to the rotation and translation of the wheel 38, the outermost part 81 of the first wall 71 is deformed by the female forming tool 55 of the wheel 38. As
30 the wheel 38 is further rotated and translated towards the third passage 53, the second tooth 62 will be introduced into the third passage 53, the first wall 71 will be deformed to obtain a shape defined by the female forming tool 55 as shown

in Fig. 7. Due to the further rotation and translation of the wheel 38, the second tooth 62 will be introduced in the third passage 53. During this process, the outermost part 82 of the second wall 71 will be deformed due to its being introduced
5 in the female forming tool 55 of the wheel 38 so as to be deformed substantially as illustrated in Fig. 7. Due to the further rotation and translation of the wheel 38, the female forming tool 55 will, until the teeth 61 and 62 are equally introduced in the passages 51 and 53, respectively, which is
10 almost the situation in a passage 54 closed prior to the passage 52, press the outermost deformed wall parts 81 and 82 together so as to ensure a suitable and tight closure of the second passage 52.

After this, the first tooth 61 will begin to be retracted
15 from the first passage 51, and when the second tooth 62 has been withdrawn from the third passage 53, the deformed wall parts 81 and 82 have met over the second passage 52 whereby this passage has been closed at this end of the filter body 31.

20 As will be seen from Figs. 6 and 7, the closure of the closed channel 52 is positioned at a small distance from the surface 34 of the filter body 31. This, however, has no effect on neither the strength of the filter body 31 nor on the filtering capability of the filter body 31. This distance is typi-
25 cally in the order of 2 mm. The difference in the appearance of the filter bodies according to the first and the second embodiments is solely visual. The filtering capabilities and the strength of the filter bodies does not depend on this difference.

30 This example has been described using a single wheel or a mould having a base describing a curved surface. However, it will increase the productivity of the manufacturing process if a number of wheels were assembled on a common shaft so as to deform and close all the relevant passages at one end of
35 an extruded body in one process. Furthermore, it will be

possible to use two such setups in a manner so that the extruded body may be deformed in both ends at the same time. This, of course, will require that the two shafts are inter-related so as to avoid that some channels are closed at both
5 ends and others not closed at all.

Figs. 8 and 9 illustrate alternative embodiments for use in the method of the invention. The mould 91 of Fig. 8 may replace the mould 20 of Figs. 3 and 5, and the mould 92 of Fig. 9 may replace the mould 38 of Figs. 6 and 7.

10 Fig. 10 is substantially identical to Fig. 6 except for the additional shape-preserving body 93. This body is inserted in order to assure that the deformed walls obtain the correct shape during deformation. If the filtering walls have a tendency to crack or break up, the body 93 and the female
15 forming tool 40 (which for illustration purposes have been removed from contacting the extruded body) will define the correct shape which the deformed filtering wall should obtain after deformation.

It may be preferred to insert a number of shape-preserving
20 bodies 93 into the filter body sample during deformation. Thus, it may be preferred to insert bodies 93 in all passages to be closed.

EXAMPLE 1: Wet deformation

In the following, the manufacture of a filter body made of
25 SiC particles is described.

The formula for the plastic material may be varied for different purposes. Presently preferred typical materials for a filter body having a suitable filtration efficiency and an acceptable pressure drop over the filter body, and which also
30 provides a good mechanical strength and good thermal prop-

erties for use on e.g. a diesel engine-driven fork lift truck are within the ranges given in the below Table 1:

TABLE 1

		wt%	such as
5	Cellulose ether *	3-10	5.2 wt%
	Poly vinyl alcohol (10% solution)	0-2	1.3
	Stearic acid **	0-2	1.3
	Ethylene glycol ***	0-4	1.0
	Ethanol	0-10	6.4
10	Water	5-15	8.1
	Coarse SiC (10-250 μ m diameter ****)	60-70	64.1
	Fine SiC (Submicron *****)	5-15	12.8
	* such as Tylose MH 300p		
	** could be replaced by other lubricants such as		
15	Microcrystalline wax (polyethylene wax or emulsion thereof) or paraffine wax		
	*** could be replaced by other plastifiers such as		
	poly(ethylene) glycol, glycerine, dioctyl phthalate or poly(propylene) glycol		
20	**** such as F180 from Arendahl Smelteverk a.s.		
	***** such as FCP 10-S from Arendahl Smelteverk a.s.		

The ingredients are weighed in separate containers. Firstly, the dry ingredients (coarse and fine SiC, cellulose ether) are mixed. Then Poly vinyl alcohol, Stearic acid, Ethylene glycol and alcohol are added and finally the water is added. After additional mixing the material is ready for extrusion.

The extrusion process is preferably performed using a standard single auger extruder.

The mixed material is fed into the feeding auger of the extruder where the material is conditioned. From here it is pressed through a noodle-die, transforming the material into "noodles" which are chopped up by a rotating knife. The material now falls into the vacuum chamber of the extruder.

From the vacuum chamber the material is transported by the main auger, which compresses and homogenizes the material. The material is pressed through the extrusion head at a pressure in the range of 15-80 bar and is received on a
5 electronically controlled table or on a table with an air cushion. The extrusion temperature is 15-55°C, such as in the order of 30°C. The bodies are cut in suitable lengths by a grinder, and are placed in a container to be used when drying the bodies.

- 10 After extrusion, the extruded bodies are dried, e.g. in a microwave oven (Husquarna QN1276F) added extra ventilation. After 15 minutes of drying at full effect (650W) and 15 minutes at half effect the bodies are dry.

The extruded body has a wall thickness of 0.5-1.0 mm, and the
15 passages thereof are quadratic having a side length of in the order of 2.5 mm.

The dried bodies are rigid and hard enough for handling and machining. The dimensions of the bodies are measured, and the bodies are machined to obtain the desired length. The cutting
20 of the bodies may be performed using a grinding plate or a diamond-coated cutting disk.

To be able to better deform the material in an end of the dried extruded body, this end is preferably soaked in a soaking liquid, such as water, water/ethanol, water/wax
25 emulsion, water/detergent or water/oil emulsions. In the present example pure water was used. The end of the body may be soaked up to 5 mm from the end of the body. After this, the soaked end is inserted into a suitable tool which re-structures the humid and deformable walls of the body. The
30 walls are restructured to meet over a passage which is to be closed.

In this example, a tool of the type shown in Fig. 5 is used. The pyramid-shaped elements have a side length of in the

order of 4 mm and a height of in the order of 4-7 mm. The distance between the individual pyramid-shaped elements was in the order of 4.8 mm.

The soaking of one end of a filter body sample may be performed by placing this end in a container containing 10 mm of the soaking liquid for 2-10 minutes, such as 5 minutes. After that, the wet end of the filter body sample is positioned on a sponge positioned in a containing 5 mm of the soaking liquid for a period of time, such as 2 to 10 minutes, preferably in the order of 5 minutes, whereafter the soaked end of the body is placed on a more dry sponge for removing excess soaking liquid for a second period of time, such as 5 to 30 minutes, preferably in the order of 10 minutes. Finally, the filter body sample is positioned on a dry surface with the soaked end pointing up for a third period of time, such as 2 to 10 minutes, preferably in the order of 5 minutes. During this period of time, the soaked end may be soaked every e.g. 5 minutes until the filter body sample has a suitable working consistency.

After the first soaking step, the filter body sample may alternatively be prepared by heating in a micro wave oven at full effect (650W micro wave power) for 3-4 minutes.

The deformation preferably takes place in a number of steps. In the first step, the walls are deformed by a deforming tool having relatively a sharp angle (see Fig. 3) of 15-30°, such as in the order of 22.5°. After this, further deformation is performed using tools having less sharp angles, such as 25-60°, preferably in the order of 30°.

Inbetween the shaping steps, the end of the filter body sample is "rolled", e.g. by using the end of a polished steel cylinder. Thus, the material of the deformed walls is kneaded together to ensure that the passages are closed and that the closures have sufficient strength.

Furthermore, to prevent adhesion of the tools to the soaked end of the body, SiC powder may be sprinkled on the soaked end. This SiC powder is preferably either fine SiC particles, such as having a particle size of 35 μm , or a mixture of 0.3-
5 2 μm SiC and 85 μm SiC.

After the restructuring, the restructured end of the body is dried whereafter a similar restructuring may be performed at the other end of the body. Care should be taken to ensure a suitable displacement so that no passages remain open in both
10 ends.

As mentioned above, blocking the passages of the filter body sample using the above described method will not substantially reduce the total area of the filtering wall in the filter body. The deformed or restructured part of the filtering wall
15 in the filter body forms a closure over the passages to be closed. This closure is made of the same material as the rest of the filtering wall of the filter body, whereby the closure will also take part in the filtration of the exhaust gas.

In the same time, both due to the not-reduced area of the
20 filtering wall and due to it being possible to form funnel-shaped entrance and exit holes for the gas, the pressure drop over the filter body according to the invention is smaller than that of a filter body incorporating typical plugs.

After restructuring of the ends of the filter body sample the
25 sample is dried as described above in an oven, whereafter the temperature is elevated to a temperature in the order of 200-600°C to evaporate the binder (pyrolysis).

After pyrolysis of the binder the bodies are sintered in e.g. an electrical heated furnace in an argon atmosphere for 15 to
30 240 minutes at temperatures above 2200°C, such as 2300°C.

EXAMPLE 2: Dry deformation

In contrast to Example 1, the deformation of the ends of the filter body may also be performed without softening the filter body by soaking in a soaking liquid.

- 5 An extruded body was manufactured as seen in Example 1 on the basis of the following slightly altered formula:

TABLE 2

	wt%	such as
Cellulose ether *	3-10	5.9 wt%
10 Poly vinyl alcohol (10% solution)	0-2	0.8
Wax emulsion (such as Licomer PE 02)	0-10	0.8
Stearic acid **	0-3	0.8
Poly ethylene glycol ***	0.8-2.54	1.3
Ethanol	0-10	3.4
15 Water	5-15	11.0
Coarse SiC (10-250 μ m diameter ****)	60-70	63.2
Fine SiC (Submicron *****)	5-15	12.2

- * such as Tylose MH 300p
- ** could be replaced by other lubricants such as
- 20 Microcrystalline wax (polyethylene wax or emulsion thereof) or paraffine wax
- *** could be replaced by other plastifiers such as (ethylene) glycol, glycerine, dioctyl phthalate or poly(propylene) glycol
- 25 **** such as F180 from Arendahl Smelteverk a.s.
- ***** such as FCP 10-S from Arendahl Smelteverk a.s.

This body was extruded as described in Example 1 under a slightly higher extrusion pressure (in the order of 10 bars higher).

- 30 The extruded body was dried in a drying cabinet having a starting relative humidity of 95% and a temperature of 80°C. The relative humidity and the temperature were lowered to 55%

and 20°C, respectively, (ambient atmosphere) during four days, whereafter the extruded bodies were dry.

After drying, as described in Example 1, the dried bodies were machined to obtain the desired dimensions.

- 5 Deforming the ends of a filter body which have not been softened naturally requires a larger force compared to a softened filter body. Thus, it may be preferred that dry-deformation of filter bodies is performed by a machine. This machine may comprise a holding means comprising a metal
10 support and a pneumatic rubber bellows which, when inflated, will hold the filter body. The deforming mould may be held by a mould moving means moving the mould toward the filter body in a linear movement. The mould in this embodiment, thus, should be a mould having a straight base, such as the mould
15 seen in Figs. 3 and 5.

The force required to close a single channel may be in the order of 50-500 grams. Thus, the force required to deform all relevant channels at the end of a filter body containing e.g. 800 channels may be in the order of 40-400 kg.

- 20 As large forces are required in order to perform the dry-deformation, it may be preferred to introduce shape-preserving means into the channels in order to prevent collapse of the filter body walls during deformation. These shape-preserving means may be suitably shaped rods introduced from the
25 side opposite to that of the mould.

The instrument performing this dry-deformation may furthermore comprise a number of sensors to ensure the desired precision of the deformation process:

- a transducer for measuring the deformation force,
- 30 - a transducer for ensuring the correct motion distance of the mould into the filter body,

- a device for measuring the height of the filter body in question, and/or
- a device for determining the outer diameter of the filter body in question.

- 5 The information from the different sensors may be fed into e.g. a PLC which then controls the deformation of the filter bodies.

- As filter bodies may be manufactured with a precision of the order of 0.5-0.1 mm, it may be preferred that the precision
- 10 of the deformation is in the same order of magnitude. Naturally, this will require a strict control with the deforming instrument. This precision may be obtained by either employing a number of suitably chosen and suitably positioned sensors. An improved precision may also be obtained by allow-
- 15 ing the deforming mould to be displaceable in order to adapt to e.g. small rotations of the filter body.

EXAMPLE 3: Comparison Example

- A comparison has been made between two filter bodies A and B. Both filter bodies have been manufactured as described in
- 20 Example 1 with the exception that B, instead of the method of the invention, has been plugged in accordance with the prior art. In B plugs of a length of 5-7 mm were introduced in each end in a checkerboard-like manner. The material of the plugs was identical to the material of the extruded body.
- 25 The two filter bodies were surrounded by an insulating material and canned in a steel can.

A test was performed in which gas at 20°C was blown through the filter bodies while the volumetric flow (m^3/s) of gas and the counter pressure over the filter bodies was measured.

The results of these tests is shown in Fig. 11, wherefrom it is clear that filter body A has the superior flow characteristics; a generally lower counter pressure.

It should be noted that the graphs of Fig. 11 have been
5 corrected to take into account the loss of filter wall of
filter body B. Thus, in the graphs of Fig. 11, the effective
filter wall area is used. Thus, the lower counter pressure
seen in this test is solely based on the funnel-shaped chan-
nel entrances and exits. If the lower filter wall area of
10 filter body B was further taken into account, an additional
2-10% higher counter pressure will be seen in B.

Thus, it is clear that the closing method of the invention renders filter bodies superior over the prior art.

Claims

1. A method for closing a passage in a filter body sample made from a solidifiable plastic or plastifiable particle-based material and comprising a plurality of coextending
5 throughgoing passages separated by common passage walls, the method comprising deforming passage walls adjacent to the passage.
2. A method according to claim 1, wherein the deformation of the walls is performed by introducing the walls into a female
10 forming tool forcing the outermost parts of the walls together.
3. A method according to claim 2, wherein the female forming tool is defined by adjacently positioned male forming tools positioned on a base of a mould.
- 15 4. A method according to claim 3, wherein the male forming tools are positioned so that each male forming tool is pressed into an individual passage coextending with the passage being closed.
5. A method according to claim 4, wherein the individual
20 passages into which the male forming tools are pressed are passages adjacent to the passage being closed.
6. A method according to claim 5, wherein the passage being closed and the individual passages into which the male forming tools are introduced have in common the walls which are
25 deformed.
7. A method according to any of the preceding claims, wherein several of the passages in the filter body sample are closed.
8. A method according to any of the preceding claims, wherein the cross-sectional arrangement of the passages is in a

number of substantially parallel rows, the passages in each row being arranged substantially equidistantly.

9. A method according to claim 8, wherein the cross-sectional arrangement of the passages is in a regular pattern, the
5 individual passages having substantially identical polygonal cross-sections.

10. A method according to any of the preceding claims, wherein the male forming tools are positioned on a substantially plane base whereby the deformation of the passage walls is
10 performed substantially simultaneously by all female forming tools on the mould.

11. A method according to any of claims 1 to 9, wherein the base on which the male forming tools are positioned is shaped substantially as part of a drum which, when rolled over the
15 filter body sample, sequentially introduces the male forming tools in the passages, whereby the walls are deformed sequentially.

12. A method according to any of the preceding claims, wherein the plurality of passages are divided into first and
20 second groups, the passages of the first group being closed at a first end of the filter body sample and the passages of the second group being closed at an opposite second end thereof.

13. A method according to any of the preceding claims, further comprising the step of solidifying the sample subsequent
25 to the deformation.

14. A method according to any of the preceding claims, wherein, prior to the deformation, a shape-preserving body is introduced in at least one of the passages to be closed, the
30 shape-preserving body being an elongated body having a cross-section substantially identical to the cross-section of the passage to be closed so that the shape-preserving body will

substantially fill the cross-section and support the passage walls during deformation.

15. A method according to claim 14, wherein the shape-preserving body is introduced from the end of the filter body
5 which is opposite to the end at which passage walls are to be deformed.

16. A method for producing a filter body for filtering flue gas, the method comprising

- 10 - producing, from a solidifiable plastic or plastifiable particle-based material, a filter body sample comprising a plurality of coextending throughgoing passages separated by common passage walls,
- 15 - closing one end of substantially each passage by deforming passage walls adjacent to the passage, and
- subsequently solidifying the plastic or plastifiable particle-based material to a porous solid material.

20 17. A method according to claim 16, wherein the plurality of passages are divided into first and second groups, the passages of the first group being closed at a first end of the filter body sample and the passages of the second group being closed at an opposite second end thereof.

25 18. A method according to claim 17, wherein the throughgoing passages of the first and second groups are closed in a checkerboard pattern so that substantially each passage of the first group has common walls solely with passages of the second group and vice versa.

30 19. A method according to claim 17, wherein the passages of the first or the second group are closed by deforming adjacent passage walls thereof by introducing the walls into a

female forming tool forcing the outermost parts of the walls together.

20. A method according to any of claims 16-19, wherein the female forming tool is defined by adjacently positioned male
5 forming tools positioned on the base of a mould.

21. A method according to claim 20, wherein the male forming tools are positioned so that each male forming tool is pressed into an individual passage coextending with the passage being closed.

10 22. A method according to claim 21, wherein the individual passages into which the male forming tools are pressed are passages adjacent to the passage being closed.

23. A method according to claim 22, wherein the passage being closed and the individual passages into which the male forming tools are introduced have in common the walls which are
15 deformed.

24. A method according to any of claims 16-23, wherein the cross-sectional arrangement of the passages is in a number of substantially parallel rows, the passages in each row being
20 arranged substantially equidistantly.

25. A method according to claim 24, wherein the cross-sectional arrangement of the passages is in a regular pattern, the individual passages having substantially identical polygonal cross-sections.

25 26. A method according to any claims 16-25, wherein the male forming tools are positioned on a substantially plane base whereby the deformation of the passage walls is performed substantially simultaneously by all female forming tools on the mould.

27. A method according to any of claims 16-25, wherein the base on which the male forming tools are positioned is shaped substantially as part of a drum which, when rolled over the filter body sample, sequentially introduces the male forming tools in the passages, whereby the walls are deformed sequentially.

28. A method according to any of claims 25-27, wherein the cross-sectional shape of the through-going passages are polygons and where the male forming tools have a cross-section being a polygon of the same order as the polygons of the cross-sectional shape of the through-going passages.

29. A method according to claim 28, wherein the polygon is of order 4.

30. A method according to claim 29, wherein at least the tips of the male forming tools are of the shape of a pyramid.

31. A method according to any of claims 16-30, wherein the particle-based material is a particle-based ceramic material, and the mean pore size of the solidified particle-based ceramic material is in the range of 10 to 100 μm .

32. A method according to claim 31, in which the mean pore size of the solidified particle-based ceramic material is in the range of 15-80 μm .

33. A method according to claim 32, in which the mean pore size of the solidified particle-based ceramic material is in the range of 20-70 μm , in particular 30-50 μm .

34. A method according to any of claims 31-33, wherein the porosity of the particle-based ceramic material is in the range of 30-90%.

35. A method according to claim 34, wherein the porosity of the particle-based ceramic material is in the range of 40-75%.

5 36. A method according to any of claims 31-35, wherein the particle-based ceramic material is made from particulate SiC having a weight average mean particle size in the range of 10-250 μm , such as in the range of 20-150 μm , preferably in the range of 30-100 μm .

10 37. A method according to any of claims 16-36, wherein the solidification of the body is performed by heat-sintering of the body.

38. A method according to any of the preceding claims, wherein the filter body sample is made by extruding the solidifiable plastic particle-based material.

15 39. A filter body of a porous solid particle-based ceramic material, comprising a plurality of coextending throughgoing passages separated by common passage walls, substantially each of the passages being closed at one end thereof by a closure formed due to the walls of the passage converging,
20 the wall thickness of the major part of the area of the closure substantially not exceeding the wall thickness of the filtering walls.

40. A filter body according to claim 39, wherein the solid particle-based ceramic material is SiC.

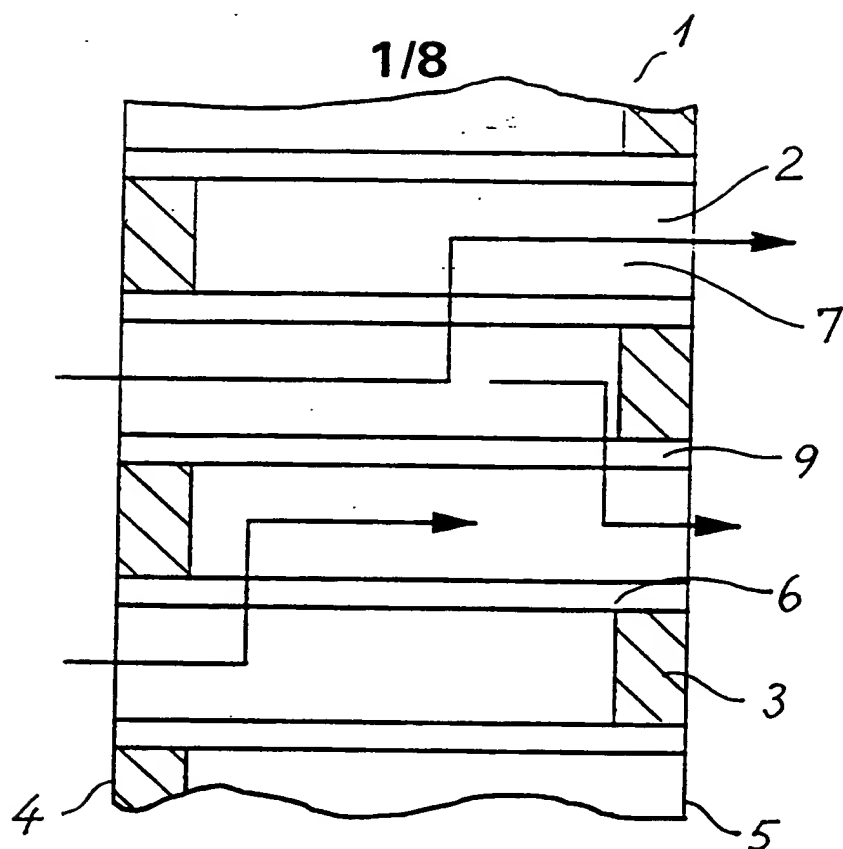


Fig. 1

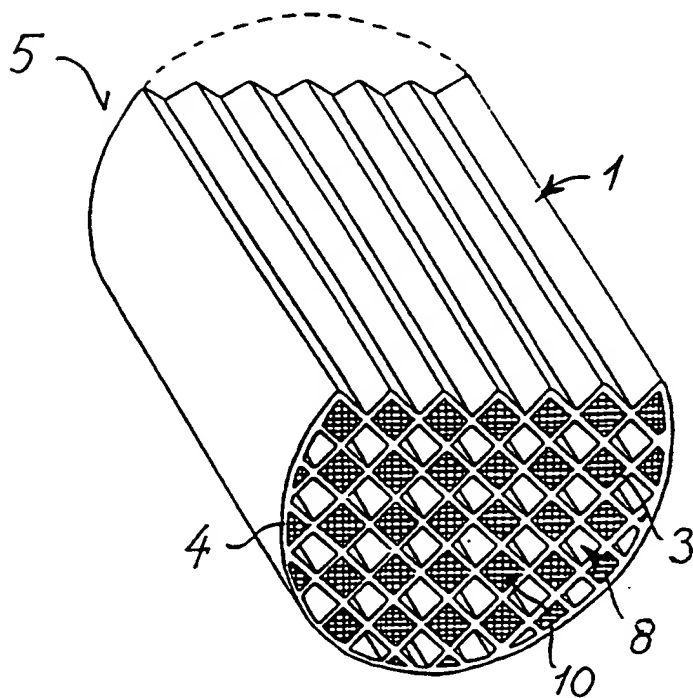


Fig. 2

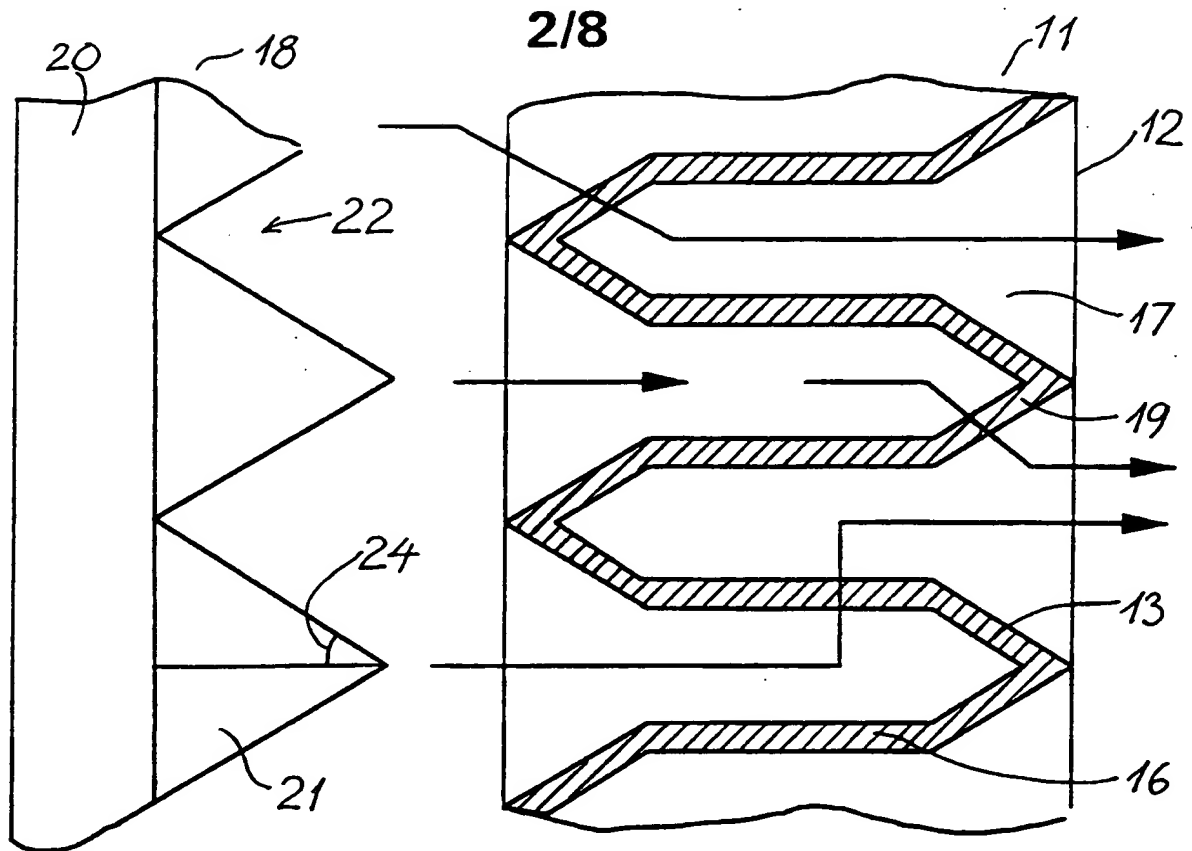


Fig. 3

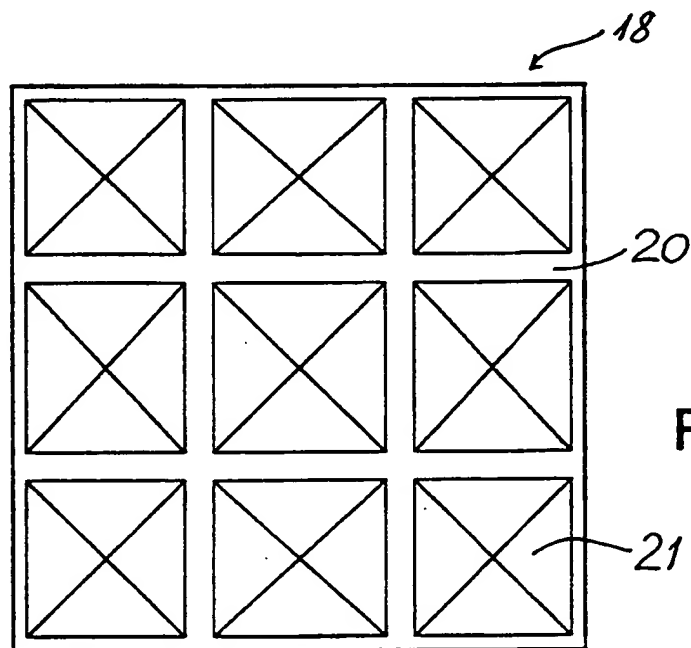
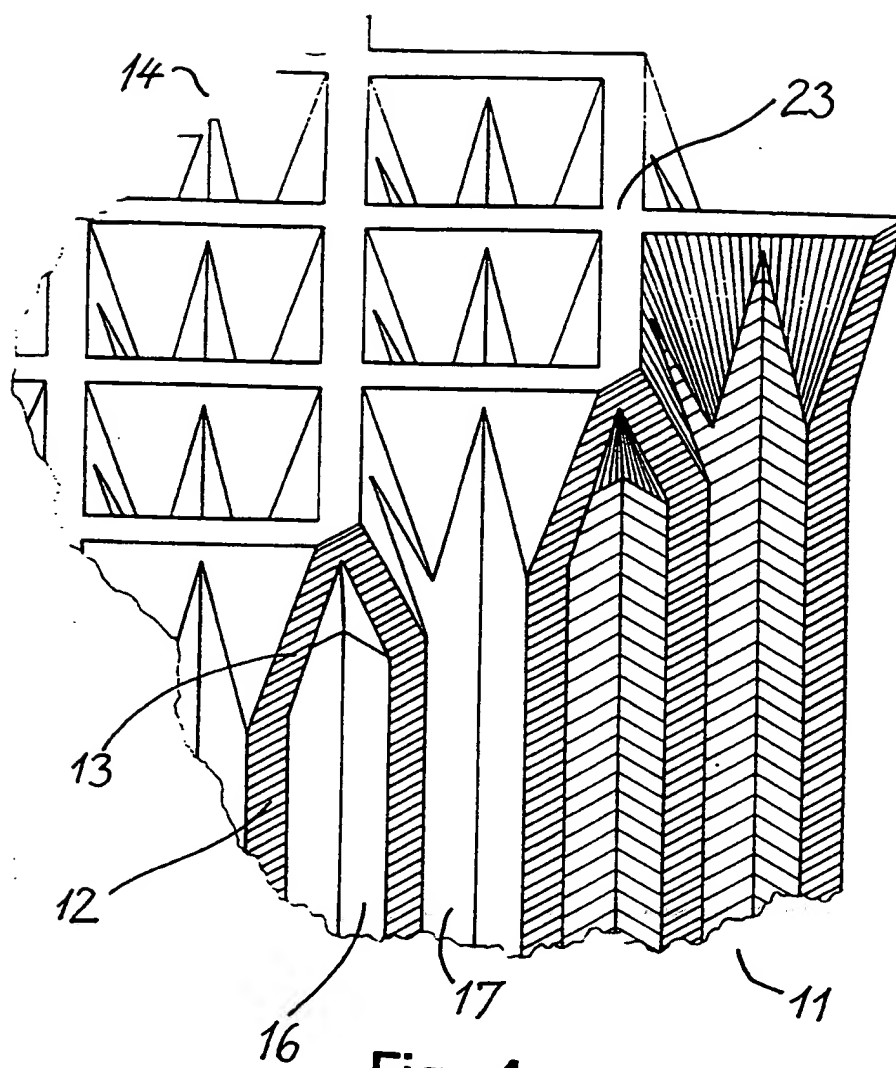


Fig. 5

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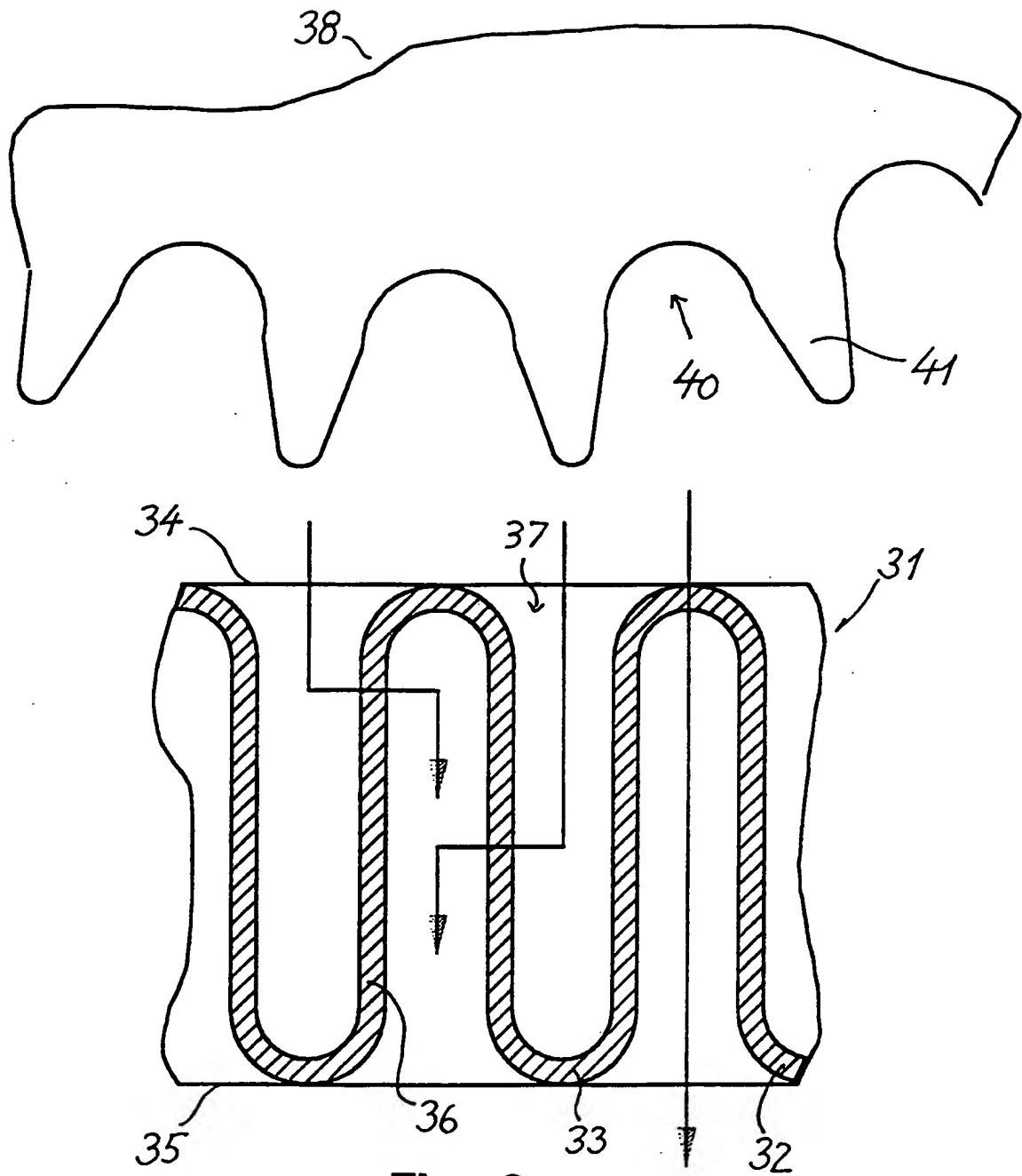


Fig. 6

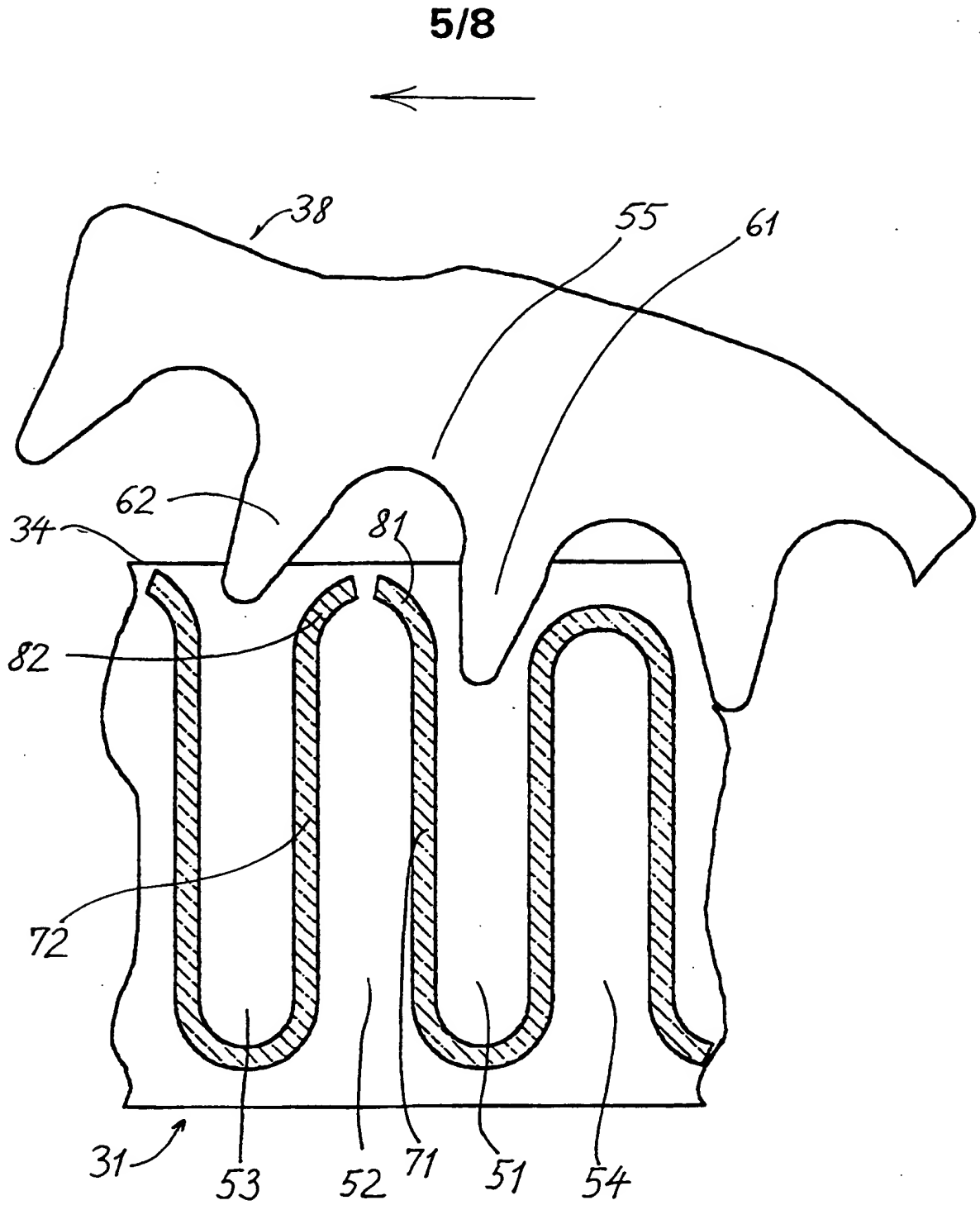


Fig. 7

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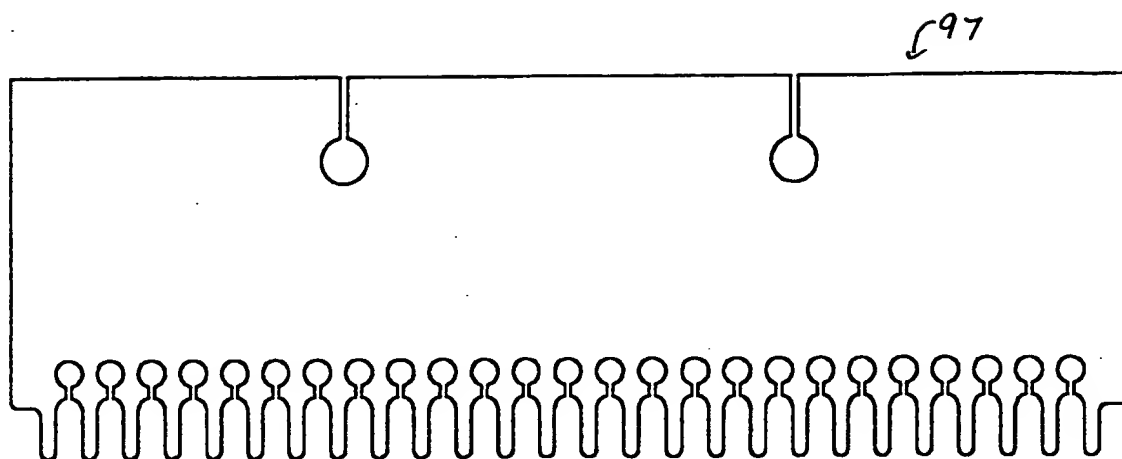


Fig. 8

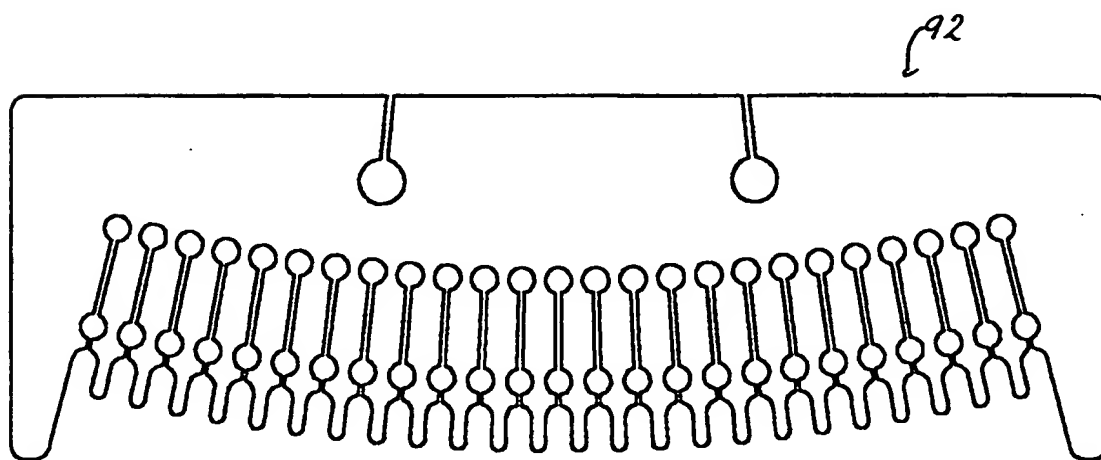


Fig. 9

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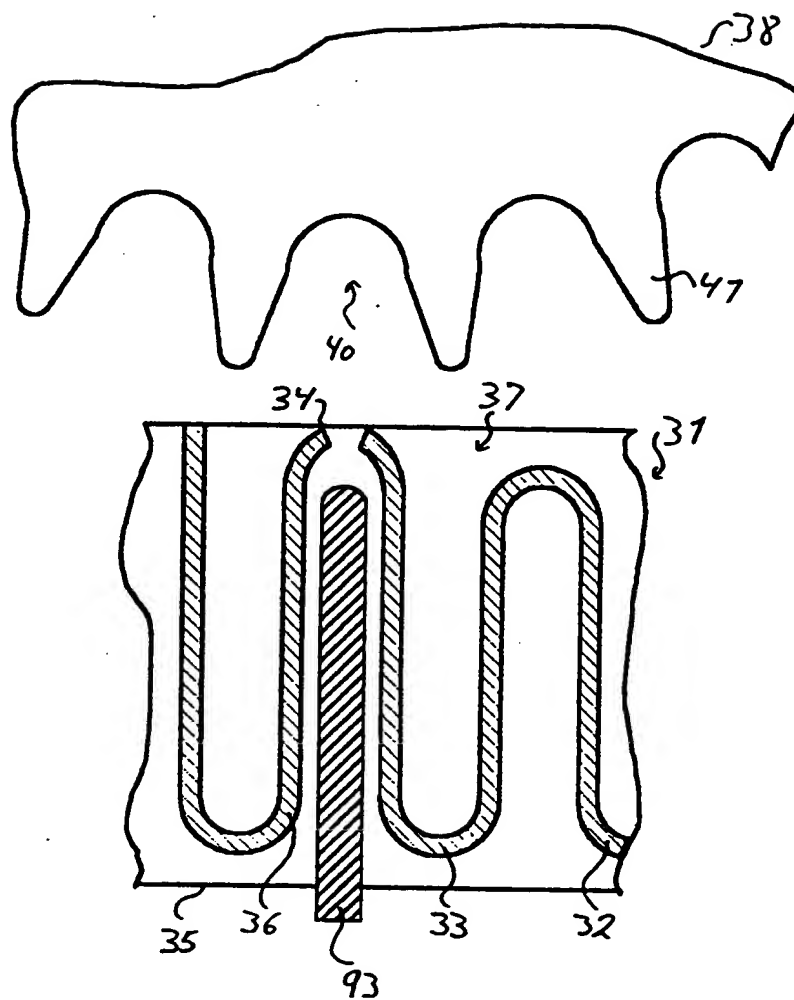


Fig. 10

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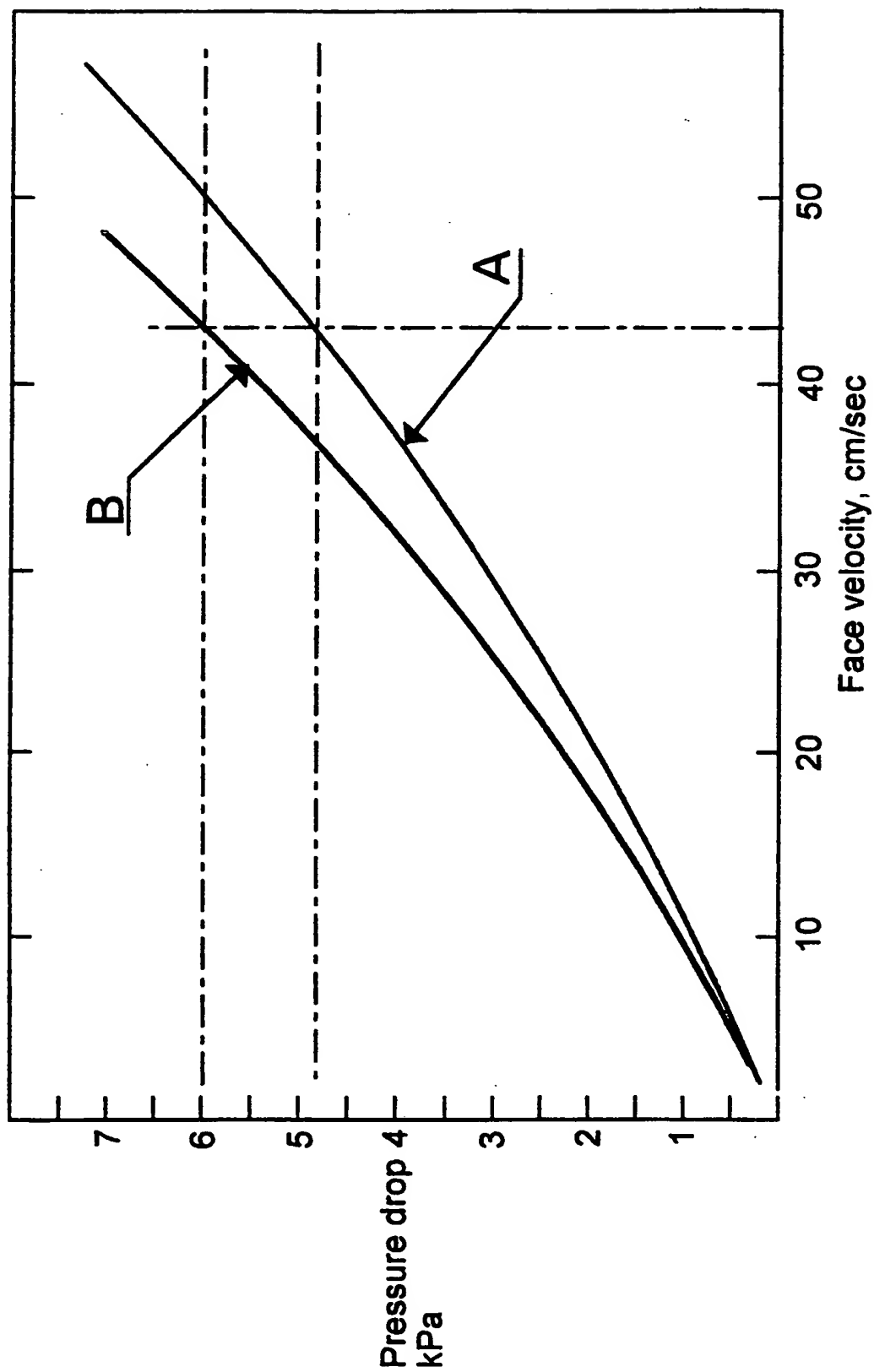


Fig. 11

INTERNATIONAL SEARCH REPORT

International application No.

PCT/DK 94/00140

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: B01D 46/10, B01D 46/24, F01N 3/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: B01D, F01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, CLAIMS, EPAT

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4662911 (TSUKASA HIRAYAMA ET AL), 5 May 1987 (05.05.87), column 5, line 12 - column 6, line 20 --	1-40
A	WO, A1, 8909648 (STOBBE, PER), 19 October 1989 (19.10.89), page 20, line 4 - line 21; page 22, line 4 - page 24, line 20 --	1-40
A	EP, A1, 0089756 (CORNING GLASS WORKS), 28 Sept 1983 (28.09.83), page 17, line 5 - page 18, line 2 --	1-40

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

21 July 1994

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/DK 94/00140

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP, A1, 0206250 (NIPPONDENSO CO., LTD.), 30 December 1986 (30.12.86), column 3, line 22 - column 4, line 39 --	1-40
A	EP, A1, 0446046 (NGK INSULATORS, LTD), 11 Sept 1991 (11.09.91), page 6, line 1 - line 2, abstract -- -----	1

INTERNATIONAL SEARCH REPORT
Information on patent family members

02/07/94

International application No.

PCT/DK 94/00140

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4662911	05/05/87	JP-A- 58161962 JP-C- 1734174 JP-B- 4024531 JP-A- 59000520	26/09/83 17/02/93 27/04/92 05/01/84
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